Macroscopic Tuning of Nanomechanics: Substrate Bending for Reversible Control of Frequency and Quality Factor of Nanostring Resonators

Scott S. Verbridge,† Daniel Finkelstein Shapiro,‡ Harold G. Craighead,‡ and Jeevak M. Parpia*†

Department of Physics and the Cornell Center for Materials Research, Cornell University, Ithaca, New York 14853, and School of Applied and Engineering Physics and the Cornell Center for Materials Research, Cornell University, Ithaca, New York 14853

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ABSTRACT

We have employed a chip-bending method to exert continuous and reversible control over the tensile stress in doubly clamped nanomechanical beam resonators. Tensile stress is shown to increase the quality factor of both silicon nitride and single-crystal silicon resonators, implying that added tension can be used as a general, material-independent route to increased quality factor. With this direct stretching technique, we demonstrate beam resonators with unprecedented tunability of both frequency and quality factor. Devices can be tuned back and forth between a high and low stress state, with frequency tunability as large as several hundred percent demonstrated. Over this wide range of frequency, quality factor is also tuned by as much as several hundred percent, providing insights into the loss mechanisms in these materials and this class of nanoresonator. Devices with frequencies in the 1−100 MHz range are studied, with quality factor as high as 390 000 achieved at room temperature, for a silicon nitride device with cross-sectional dimensions below 1 μm, operating in a high stress state. This direct stretching technique may prove useful for the identification of loss mechanisms that contribute to the energy balance in nanomechanical resonators, allowing for the design of new devices that would display higher quality factors. Such devices would have the ability to resolve smaller addendum masses and thus allow more sensitive detection and offer the potential for providing access to previously inaccessible dissipation regimes at low temperatures. This technique provides the ability to dramatically tune both frequency and quality factor, enabling future mechanical resonators to be used as variable frequency references as well as variable band-pass filters in signal-processing applications.

Nanomechanical resonators continue to be of interest to a diverse audience due to their utility as tools for a range of applications including signal processing,1 mass sensing,2,3 and observation of quantum effects in mechanical systems4 as well as the promise of new materials to provide access to previously inaccessible size scales and sensitivities.5 Small mass, high frequency, tunable frequency, and high quality factor are all desirable features. Typically high frequency is achieved in part by choosing small resonator size, while quality factor is usually diminished with shrinking device size.6 Tuning of lower frequency micromechanical devices (~10 kHz operating frequencies) by as much as 300% has been accomplished using a scanning tunneling microscope tip to provide both a local drive and mode constraint,7 but this technique would be difficult to scale down in size to achieve higher-frequency tunability. The ability to reversibly tune frequency in nanomechanical devices of a given size, operating at radio frequencies, has previously been achieved by tuning stress capacitively,8,9 as well as electrothermally.10 Due to leakage currents, capacitive lock-in, as well as thermal limitations, these techniques are typically only successful in tuning frequency by a few percent. Reversible tuning of quality factor by more than a few percent within the linear drive regime has not previously been demonstrated, and the precise nature of the energy dissipation in this class of resonators, which determines the quality factor, is still not well understood.

We have previously demonstrated that tensile stress can be used as a parameter for achieving increased frequency as well as increased quality factor.11 In this Letter we demonstrate a technique by which beam tension can be continuously and reversibly controlled by bending of the nanoresonator chip, similar to a technique used in molecular electronics for mechanically controlling break junction gap distance.12
With this technique, we demonstrate unprecedented frequency and quality factor tunability for silicon nitride beams. We also show preliminary results on single-crystal silicon beams, in which stress is demonstrated to positively affect quality factor. Finally, we argue that surface losses are ultimately limiting the quality factor in the high stress devices considered. This is in agreement with results of other groups, who have studied the effects of surface treatments and annealing on quality factor. But our work demonstrates that while surface losses may be important for nanoscale resonators, they are not necessarily the dominant loss mechanism for doubly clamped beam resonators, since altering the stress state, thought to affect mechanical clamping losses, is demonstrated here to have a dramatic effect on the quality factor of the devices studied, in some cases reducing mechanical dissipation by almost an order of magnitude.

Devices are patterned lithographically, either optically or with an electron beam, on a substrate consisting of a silicon nitride or single-crystal silicon device layer and a sacrificial silicon dioxide layer on a silicon handle wafer, with plasma etch and wet release steps the same as those previously reported. Handle wafers are either 500 or 275 µm thick silicon wafers for the silicon nitride devices or 1 mm thick silicon wafers for the single-crystal silicon devices. Silicon nitride devices are all between 105 and 120 nm thick, and silicon devices are all 205 nm thick, unless otherwise noted. Widths are all in the 400 nm to 1.0 mm range, unless otherwise noted (over this range, width was not observed to be a critical parameter for the results that follow). Measurements are taken in vacuum, at pressures below \(10^{-3}\) Torr, to eliminate viscous damping. An amplitude-modulated blue laser (405 nm, PicoQuant laser head and modulator) is used to thermally stimulate beam resonance, while a continuous wave red laser (633 nm HeNe laser) is used for interferometric detection, with the resonator and underlying substrate forming a Fabry–Perot interferometer. A schematic for this optical drive and detection setup is shown in Figure 1. Devices are stretched reversibly using an apparatus which either clamps a chip at its edges and induces curvature by application of a force in the center (as shown in the schematic in Figure 1) or which clamps the chip at both edges and moves one edge relative to the other. The later method is used to both stretch and unstretch devices, so that tension can either be added or removed. Stretching is controlled in the former case with a small screw contained within the vacuum chamber or in the latter case with a screw coupled to a mechanical vacuum feedthrough.

For uniform substrate bending, it is expected that a surface nanobeam should experience a strain given by

\[
\frac{\delta l}{l} = \frac{3t \delta y}{R^2}
\]

where \(t\) is wafer thickness, \(R\) the distance between the clamping points, and \(\delta y\) is the deflection distance from horizontal at the center of the chip. For a chip thickness of 275 µm, chip size of 1 cm, and a center deflection of 3 × the chip thickness, the resulting strain is \(6.8 \times 10^{-3}\). For a Young’s modulus of \(\sim 200\) GPa assumed for both low and high stress silicon nitride,\(^{11,19}\) this implies that an initially stress-free silicon nitride beam on a chip stretched in the way described would have a resulting stress of \(\sim 1400\) MPa. This compares well to the 1200 MPa tensile stress previously reported for high stress nitride beams yielding elevated quality factors\(^{11}\) and implies that the described stretching technique should allow low stress nitride beams to be pushed into a higher stress, higher \(Q\) regime.

Because of the nonuniform curvature resulting from the stretching apparatus, small amounts of slipping at the clamping points, and slipping and sticking of sliding parts of the mount, it is difficult to precisely correlate screw turns with chip bending. Therefore we use resonant frequency to accurately track beam stress. It is known from continuum mechanics that the fundamental resonance frequency of a doubly clamped beam of variable tensile stress should follow

\[
f = \left(1.03 \frac{t}{L^2} \sqrt{\frac{E}{\rho}} \sqrt{1 + \frac{\sigma L^2}{3.4E t^2}} \right)
\]

for thickness \(t\), length \(L\), Young’s modulus \(E\), density \(\rho\), and tensile stress \(\sigma\), where the term in parentheses is the resonant frequency of an unstressed beam. With this relation in mind, it is possible by monitoring beam frequency, to ensure that the stress state is in fact being affected by the chip bending. By monitoring frequency, we can also determine to what extent the devices, once relaxed, return to their initial stress values and quality factors, indicating that the effects of added
stress on quality factor are in fact reversible. In the following, we will describe the effects of stretching on silicon nitride resonators with two different inherent stress values, as well as single-crystal silicon resonators.

Devices were first fabricated in low-stress silicon nitride, with tensile stress in the 150–200 MPa range (measured with a wafer bow technique, using a Flexus tool). Chips with beams made of this low-stress silicon nitride were bent until frequencies of individual devices were observed to have shifted to values just below the values at which similar chips fractured. Quality factors over this range of bending were measured and are shown in Figure 2 for devices 5 and 10 um in length (cross-sectional dimensions will be left out of the text for ease of reading but are included in the figures). The 5 um long device initially had a frequency of 14.6 MHz, and a Q of 1200. At the highest stress value attained for this device, the frequency was 35.5 MHz, and Q was 6700. For comparison, a device of this size made from the higher stress silicon nitride (~1200 MPa inherent film stress, studied in our previous work) typically has a frequency of ~60 MHz, with a Q of ~12000. For the 10 um device, frequency and Q in the low stress state were 8.6 MHz and 3400, and in the high stress state increased to 23.8 MHz and 23000 (an increase in Q of almost 600%), as compared with the values of 30 MHz and 30000 expected for a high-stress device of this size. Frequency and quality factor values were observed to return to their initially low values when added stress was relieved. It should be noted that the low-stress devices studied were not tuned to the point at which their Q equaled that of high-stress devices of the same size, due to chip fracture issues. While these traces do extrapolate fairly closely to the values expected for high-stress devices, it might be that the quality factors do not reach values that are quite as high as in the high-stress film case. This is not surprising, as the low- and high-stress nitride films differ not only in their stress-states but also in chemical composition (the low-stress film has a higher silicon content) and surface roughness (the low-stress film has a higher surface roughness).

It is clear that adding stress to low-stress nitride resonators increases both frequency and quality factor for these devices. But in none of these experiments did the quality factors exceed those attained with the higher film stress devices of the same dimensions, previously reported. So it is interesting to consider whether stretching, or perhaps even unstretching (to relieve stress) the already high-stress devices will improve their quality factors. Experiments to investigate this regime were carried out on a number of high-stress, high-Q devices, made from a film with measured inherent stress of 1200 MPa. The results obtained for a pair of devices are shown in Figure 3. A 30 um long device was examined with frequency and quality factor of 9.3 MHz and 105000 as fabricated. The device chip was stretched until it broke. While the frequency was observed to increase by ~15%, no significant change in quality factor was observed. Similar results have been observed for up to 25% increases in frequency, which has been the most “tuning” that has been observed in these already-high-stress devices.

A slightly higher Q device, 40 um in length, was studied, with results shown in Figure 3. This device had as-fabricated frequency and Q of 6.9 MHz and 160000. The tension of this device was tuned in both directions. Frequency was varied between 1.5 and 8.5 MHz, with the quality factor varying between 25000 and 160000 over the range of tension accessed (similar to the relative change in Q that resulted from stretching the low-stress devices). It should be noted that some systematic decrease in Q was observed over the course of this experiment, which was not related to stress, with the final quality factor value (after relaxing the stress back to the as-fabricated value) having fallen to 125000 at 6.9 MHz, compared to the initial value of 160000. This decrease is thought to be related to some contamination produced by the actuation of the stretching apparatus, as well as a systematic degradation in quality factor over time that was observed in many devices. We will come back to this point later. It should also be pointed out that at sufficient “unstretching”, the resonator studied would be expected to enter a region of compression rather than tension. Given eq 2 for the resonance frequency of a doubly clamped beam, however, we would expect a stress-free beam of the dimensions studied here to have a resonance frequency of ~0.7 MHz, well below the lowest frequency of 1.5 MHz observed for this device. Therefore it is believed that this

Figure 2. Results of added stress on low-stress silicon nitride devices. A 5 um × 500 nm × 110 nm device with initial f and Q of ~14.6 MHz and 1200 was stretched to an increased f and Q of ~35.5 MHz and 6700. A 10 um × 1 um × 110 nm device, with an initial f and Q of 8.6 MHz and 3400, was stretched to an increased f and Q of 23.8 MHz and 23000. The arrows indicate the direction of the experiments in which stress was added to increase both frequency and quality factor.
device is in the tension regime over the entire range of the experiment. The overall picture that emerges from this experiment is a quality factor that increases approximately linearly with frequency as stress is added, tapering off at higher stresses. The highest quality factor values attained occur in the neighborhood of the stress value corresponding to the inherent value in the higher stress silicon nitride film (≈1200 MPa), with no observed increase in $Q$ resulting from increasing stress beyond this value.

It was previously argued\(^\text{11}\) that our high-stress devices were significantly closer than previous devices of this scale to being limited by thermoelastic losses. We present here, however, evidence that the thermoelastic limit has yet to be attained in these devices. To show this, we consider the standard equation for quality factor determined by thermoelastic losses for bending-dominated resonators:  

$$Q_{\text{TED}} = \frac{c_v}{E \alpha^2 T} \left( \frac{6}{\xi^2} - \frac{6 \sinh \xi + \sin \xi}{\xi^3 \cosh \xi + \cos \xi} \right)^{-1}$$  

with 

$$\xi = \frac{t}{\sqrt{\frac{\rho \alpha \rho}{2k}}}$$

where $c_v$ is specific heat per unit volume, $E$ is Young’s modulus, $\alpha$ is the coefficient of linear expansion, $t$ is resonator thickness, $\rho$ is density, $c$ is specific heat per unit mass, $k$ is thermal conductivity, and $T$ is temperature. We note, as a caveat, that we are actually closer to the stretching rather than the bending limit for our high-stress devices. The above expression predicts a strong dependence on thickness. We fabricated much thinner high-stress silicon nitride devices, for comparison with the previously reported thicker devices. Devices of 45 nm thickness were studied, with resulting quality factors of 26000 for the 27 MHz devices measured. This is quite close to the values obtained for the 110 nm thick devices, whereas the above-quoted model would predict that the thinner devices studied would have a $Q$ almost an order of magnitude higher than the thicker devices. It was also previously noted\(^\text{11}\) that quality factors in these high-stress devices depend inversely on frequency, as would be expected for devices in the thermoelastic limit. In our current work, however, we are able to decouple device length and device frequency in a manner that was not previously possible. The result is a demonstration that for a high-stress device of a given size, increasing device frequency by adding more stress does not lead to the decrease in quality factor that would be expected for thermoelastically limited devices. It is therefore clear that although our high-stress devices might be closer than other resonators of this scale to the thermoelastic limit, other dominant loss mechanisms exist which must be alleviated before this internal limit can be reached.

In Figure 4, we summarize the previously described tensioning measurements for silicon nitride devices, showing on a single plot the dependence of quality factor on frequency for a set of devices of varying lengths, as well as individual devices whose stress states were varied as previously described. The black squares represent the dependence of quality factor on frequency for high-stress devices each of a different length. The colored symbols, from low to high frequency, represent 40 μm (high-stress film), 10 μm (low-stress film), and 5 μm (low-stress film) long devices subjected to varied stresses. The three larger squares represent the resonators from the high-stress fixed length data set that are the closest in length to the three devices whose stresses are tuned. This representation makes it clear that as stress is increased in the stress-tuned devices, the quality factor—frequency relationship approaches the length-dependent behavior demonstrated by the as-fabricated high-stress resonators. In the case of the 40 μm device, the variable-stress data actually crosses the fixed-length data set, because stress in this device was tuned in both directions around the high-stress value. This summary representation is consistent with the interpretation that quality factor increases with stress,
tapering off at a value corresponding to the quality factors previously achieved with the high-stress silicon nitride film.

Experiments were also carried out on single-crystal silicon resonators. Silicon devices 5 μm in length were fabricated and studied using the techniques described. In one batch of these devices, as-fabricated $Q$ values were in the range $12000–13000$ at $\sim 36$ MHz. Under applied stress, we observed an increase in $Q$ to the range $15000–16000$ at $\sim 42$ MHz, representing an increase of $\sim 20\%$ in both frequency and quality factor. In another batch, the as-fabricated $Q$ values were around 9000 at 36 MHz, with $Q$ values increasing to $\sim 12000$ at 50 MHz under applied stress, representing increases of $\sim 40\%$ in both of these values. These latter devices were oxygen cleaned before operation, which resulted in decreased baseline $Q$ values as compared with the former batch. We were unable to stretch these devices much further due to the tendency of the thicker substrate (1 mm thick) to break in the $20–40\%$ tuning range.

It should be noted that quality factors of both silicon nitride and silicon devices were observed to degrade substantially over time, anywhere from 10 to 50% over a period of several days. Furthermore, the degradation observed was in many cases reversed by treatments that are thought to have helped return the surface state to an as-fabricated condition. It was previously mentioned that one silicon nitride device had a quality factor that fell from 160000 to 125000 over the course of a stretching experiment. Following a 1 h bake at 540 °C on a hot plate (under ambient air in a fume hood), a slightly higher than initial $Q$ value, $\sim 180000$ was attained for this device, indicating that the observed decrease in $Q$ was likely contamination-related. This reversal of the observed quality factor degradation in the silicon nitride devices accomplished by baking was most pronounced for the longest, and therefore highest, quality device that we studied.

A 75 μm long device was observed over a period of 3 days. This device had a resonant frequency of 3.7 MHz and an initial quality factor of 360000 postfabrication. After 2 days in vacuum, the quality factor had decreased to 250000. A 1 h bake at 540 °C was carried out for this device, resulting in a quality factor that was temporarily increased to a value of 390000, an increase of approximately 50% compared to the degraded value. The quality factor then began to decrease with time, as in the first time trace, shown in Figure 5. Also shown in Figure 5 is the Lorentzian response of this elevated-$Q$ resonance, as well as the response of the fifth harmonic of this device, at 22.1 MHz, both measured soon after the bake. As mentioned in our previous work, the quality factors of the harmonics of devices of a given length do not fall off as quickly with frequency as do quality factors of successively shorter length devices.13 Hence in considering harmonics, higher values of the product of frequency and quality factor are accessible. This product of frequency and quality factor, $fQ$, is a useful figure of merit for resonators, because frequently applications for these devices require high frequency as well as high quality factors, while there is typically some tradeoff between the two, with higher frequencies often being associated with lower quality factors.6 Furthermore, for internal dissipation mechanisms such as thermooelastic dissipation, phonon–electron scattering, and phonon–lattice relaxation, there is a limiting value of $fQ$ that can be attained for any resonator made from a given material, and hence it is interesting to consider how close to these theoretical limiting material values a given resonator can be made. The 22.1 MHz harmonic of this device has a quality factor of 170000, yielding an $fQ$ product of $3.75 \times 10^{12} \text{ s}^{-1}$, among the highest values reported. Nanomechanical devices with such high-quality factors at these frequencies, and at room temperature, could prove exceedingly useful for experiments at low temperature, where some of the important loss mechanisms, such as surface dissipation, would freeze out, whereas external sources of dissipation such as clamping losses might remain important. Such devices, which could conceivably have $Q$ values above a million at radio frequencies and at low temperatures, would be valuable for investigations into quantum mechanical effects such as back-action cooling,4 ground state freeze-out,21 and preparation of mechanical superposition states.22

Silicon devices also showed some decay in quality factor over time. Devices from the first referenced silicon batch demonstrated quality factors that were reduced to the range $8000–9000$ after a few days, having started with $Q$ values in the range $12000–13000$. Subsequent baking of such devices, which reversed the degradation observed in the silicon nitride devices, did not improve the $Q$ values of these silicon devices. Short (10 s) dips in diluted hydrofluoric acid (BOE 6:1) did improve the quality factor of these devices, resulting in increases in $Q$ of about 10%, indicating that perhaps a surface oxide (removed by the hydrofluoric acid and perhaps held off in part by a resulting hydrogen termination) is related to the observed quality factor degradation for the silicon devices, consistent with previously reported results.14 Silicon devices with degraded quality factors did continue to show significant improvements in quality factor when stretched. Devices from the latter batch
mentioned, which showed initial baseline quality factors in the 9000 range around 36 MHz, had degraded to values closer to 7000 after 5 days in laboratory air. When the devices were stretched to 52 MHz, one such set of devices showed $Q$ values that had increased to ~8500. Longer devices in this degraded batch demonstrated similar improvements in quality factor with added tension, with 10 μm long devices increasing from 7500 at 12 MHz to ~21000 at 24 MHz.

These observations on quality factor degradation are consistent with previous reports that quality factors of silicon devices seem strongly affected, even at low tension, by surface-related losses. We argue that although this is likely the case, tension is also a contributing factor for all of the devices we have considered, including those made from single-crystal silicon. However, further experiments should be carried out to more systematically study the differences in the nature of the surface-related losses between silicon and silicon nitride. A further study of devices of a range of materials in which both surface treatments and tensioning are carried out in conjunction would provide valuable insight into this interesting problem. Studies in which treatments such as annealing can be carried out in situ and in ultrahigh vacuum, so as to maintain pristine surface conditions for an extended period of time (avoiding the $Q$-degradation that certainly occurs between our hot plate bake step and eventual measurement after pump-down), could also result in significant improvements in quality factor for these high-stress devices.

The precise source of the increased energy confinement resulting from addition of tension, resulting in increased quality factor, remains unknown at this point. It was mentioned in our previous work that clamping losses were a likely culprit as a source of energy dissipation which could be affected by the resonator stress state, although this hypothesis was not accompanied by a theoretical model. While we remain unable to reconcile this hypothesis with theoretical treatments of clamping losses that exist in the literature, there does exist a body of evidence indicating that transmission of energy from a resonator to its supporting elements can be a significant factor in determining quality factor. The sharpness of the discontinuity between the resonator edge and base (curving smoothly into the perpendicular base edge vs a sharp right angle), has been demonstrated to significantly alter $Q$, by nearly a factor of 2, in otherwise similar cantilevers, indicating that the flow of acoustic energy into the support is a factor that is amenable to alteration. In our own previous work, it was observed that the quality factor for high-stress devices depended nearly linearly on inverse frequency, or in other words, dissipation depended linearly on frequency. At higher frequencies, however, the dependence of dissipation on frequency appeared to be diverging from this line. It was thought that this was due to the increased importance of clamping losses in these shorter, higher frequency devices. To further examine this possibility, we fabricated three parallel sets of resonators, with significantly different support compliances. Release etch steps of 5, 15, and 20 min were carried out for these three sets of devices, resulting in string resonators supported by overhanging membranes (~500 nm, 1.5 μm, and a 2 μm long, respectively, and in all cases much wider than the resonators.)

Figure 5. Observed quality factor degradation with time in our highest-$Q$ device, 75 μm × 500 nm × 105 nm, with improvement resulting from a 1 h bake at 540 °C. Also shown are the postbake Lorentzian responses for this device, with a record $Q$ of 390000 at 3.7 MHz, and its fifth harmonic with a $Q$ of 170000 at 22.1 MHz.
from either silicon nitride or single-crystal silicon, with a frequency and quality factor of these resonant devices, made by several hundred percent. Stress was used to increase the quality factor that can be tuned continuously and reversibly clamped nanomechanical resonators with frequency and on these sorts of resonating systems.

Acoustic properties of the supporting membrane are explicitly its effects on acoustic losses to the environment. Furthermore, tension that depends inherently on the tension, rather than tensile stress is reduced. But it remains unclear whether there to the overhang length, become more pronounced as the overhang membrane, even for devices that are long compared to a thin supporting membrane. Ultimately, surface-related losses are thought to be currently limiting the quality factors for the high-stress, high-\( Q \) resonators studied. With appropriate surface chemistries, and perhaps operation at low temperatures, it is hoped that these high tension devices will provide access to previously inaccessible dissipation regimes, with \( Q \) values above a million at radio frequencies not far out of reach.

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